

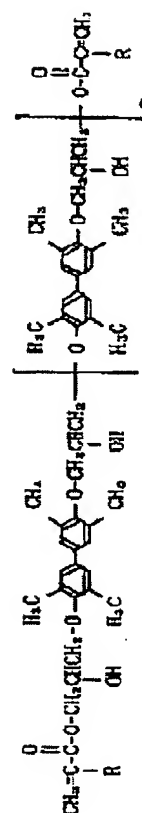
NOVEL HEAT-RESISTANT RESIN

Patent number: JP9157340
Publication date: 1997-06-17
Inventor: KUKI YUKIHIRO; TAKENAKA NAOI
Applicant: KYOEISHA CHEMICAL
Classification:
- international: C08F299/00; C09D4/02; C08F299/00; C09D4/02;
 (IPC1-7): C08F299/00; C09D4/02
- european:
Application number: JP19950345682 19951208
Priority number(s): JP19950345682 19951208

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Abstract of JP9157340

PROBLEM TO BE SOLVED: To obtain a novel heat-resistant resin which has a high glass transition temp. and low moisture absorption and is less likely to create stress and tough by using an epoxy (meth)acrylate resin (BE) having a biphenyl skeleton as a functional polymer material. **SOLUTION:** The BE resin is represented by formula I (wherein R represents H or methyl; and (n) is an integer of not less than 0) and prepd. by a general epoxy synthetic process. Specifically, it is easily prepd., in the absence of a solvent, by reacting an epoxy resin, with both terminals being glycidyl etherified, with (meth)acrylic acid in the presence of a catalyst (e.g. a quaternary salt or a Lewis base with specific examples thereof including triethylbenzyl chloride) and a polymn. inhibitor (e.g. hydroquinone) at 90 to 100 deg.C for 12 to 15hr. The BE resin may be used, as a photosensitive material, either alone or in combination with other base resin (s) and a reactive diluent for applications such as various coating agents (particularly applications where heat resistance is required), UV coating materials, or resists.



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(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平9-157340

(43) 公開日 平成9年(1997)6月17日

(51) Int.Cl. ⁸	識別記号	序内整理番号	F I	技術表示箇所
C 0 8 F 299/00	MRV		C 0 8 F 299/00	MRV
C 0 9 D 4/02	P D Y		C 0 9 D 4/02	P D Y

審査請求 未請求 請求項の数1 F D (全 6 頁)

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(54) 【発明の名称】 新規耐熱性樹脂

(57) 【要約】

【課題】 高いガラス転移温度を有し、低吸湿性で、しかも応力の発生しにくい強靱な高機能性エポキシ（メタ）アクリレート樹脂を提供する。

【解決手段】 下記式（1）で表されるビフェニル骨格を有するエポキシ（メタ）アクリレート樹脂：

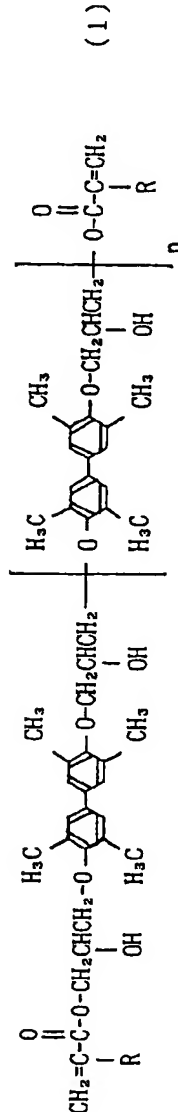
【化1】（ただし、式中のRはHまたはCH₃を表わし、nは0以上の整数を示す。）

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【特許請求の範囲】

【請求項1】 下記式(1)で表されるビフェニル骨格を有するエポキシ(メタ)アクリレート樹脂:

【化1】



(ただし、式中のRはHまたはCH₃を表わし、nは0 40 以上の整数を示す。)

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、耐熱性、感光性および低吸湿性に優れた新規なエポキシ(メタ)アクリレート樹脂に関し、特にソルダーレジストやメッキレジスト用のベース樹脂や各種コーティング用樹脂として有用なものである。さらに、本発明のエポキシ(メタ)アクリレート樹脂は、これらに限らず、単独はもとより、必要に応じて各種感光性を有する化合物とブレンドして使

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用することによって、熱的、化学的に安定な優れた高分子材料としてさまざまな用途に利用できるものである。

【0002】

【従来の技術】従来、エポキシアクリレート樹脂は、各種コーティング材としての感光用材料、光学用材料、歯科材料、高分子の架橋剤など、幅広い分野で用いられてきた。しかしながら、近年これらの応用分野における要求特性の高度化に伴い、機能性高分子材料として求められる物性はますます厳しくなっている。かかる物性として、例えば、耐熱性、耐候性、低吸湿性、高屈折率、高破壊靱性などが求められているが、これまでのところ、これらの要求物性は必ずしも満足されてきたわけではない。

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【0003】現在用いられている感光用材料の分野の中で、最も汎用的に用いられているエポキシ(メタ)アクリレートとしては、ビスフェノールタイプのものが挙げられる。このビスフェノールタイプのエポキシアクリレートは、低分子量タイプについては(例えばビスフェノールAジグリシジルエーテルジアクリレート)、比較的高いガラス転移温度を有するが、吸水率が高いうえに内部応力が発生しやすいという欠点がある。吸水率を抑え、応力の発生を抑制するためには、一般的には分子量を大きくし架橋密度を下げればよいが、逆にガラス転移温度が低下してしまうため、かかる方法にも限界がある。また、その他のタイプのエポキシアクリレートについても上記と同様に物性に関して一長一短があるのが現状である。かかる現状に鑑み、最近、耐熱性や吸水率を改善するような種々のエポキシ樹脂原料が開発されつつあるが、いずれも耐熱性、吸水率および応力の低減のすべてを満足できるまでには至っていない。

【0004】

【発明が解決しようとする課題】本発明は、かかる従来技術の問題点を解消するために創案されたものであり、その目的とするところは従来技術では達成できなかった高い要求特性を全て満足できるような新規なエポキシ(メタ)アクリレート樹脂を提供することにある。前記要求特性としては、高い熱安定性が挙げられ、そのためには第一に高いガラス転移温度が必要となるが、これについては、樹脂の架橋密度を上げることによって達成することができる。また、ガラス転移温度を高くすると、高温での安定性は保たれるが、この際、樹脂内部に蒸発成分が混入するとクラックの原因になる。特に、樹脂の吸水率が高いと自然に蒸発成分を含んでしまうため、その傾向が強くなる。従って、よほど厳しい湿度管理をしない限り高い吸湿性樹脂は使用できなくなり、熱安定性には低吸湿性が必要不可欠となる。さらに、熱膨張による内部応力が大きいほど、もろくなるため、応力の低減も重要な要素である。

【0005】上述のように、ガラス転移温度を上げるためには、一般に樹脂の架橋密度を上げることになるが、

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架橋密度を上げると、吸水率が上昇する傾向が認められる。吸水率を抑制しながらガラス転移温度を上げる手法として、嵩高い置換基（例えば多環式芳香環）や配向性を有する骨格を導入することが知られている。しかしながら、このような樹脂は応力が発生しやすいため、堅くてもろい。従って、高いガラス転移温度を有し、低吸湿性で、しかも応力の発生しにくい強靱な高機能性エポキシアクリレート樹脂の開発が要望されている。

【0006】

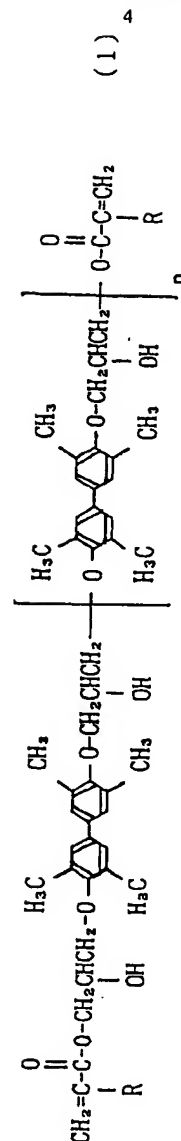
【課題を解決するための手段】本発明者らは上記目的を達成するために鋭意検討した結果、下記式（1）に示すエポキシ（メタ）アクリレート樹脂が、高いガラス転移温度を有し、低吸湿性で、しかも応力の発生しにくい強靱な高機能性エポキシアクリレート樹脂であることを知見し、本発明の完成に至った。

【0007】即ち、本発明は下記式（1）で表されるビフェニル骨格を有するエポキシ（メタ）アクリレート樹脂である：

【化2】

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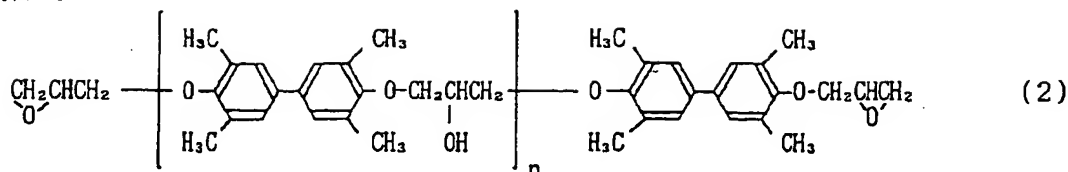
（ただし、式中のRはHまたはCH₃を表わし、nは0以上の整数を示す。）

【0008】

【発明の実施の形態】本発明のエポキシ（メタ）アクリレート樹脂は、例えば下記式（2）で示される、一般のエポキシ樹脂合成法により得られる、両末端がグリシジルエーテル化されたエポキシ樹脂とアクリル酸またはメタクリル酸を触媒と重合禁止剤の存在下で、90～100℃で12～15時間反応させることにより無溶剤下で容易に得ることができる。触媒としては、4級塩（例えば、テトラアルキルアンモニウムハライドやジハロトリフェニルホスホランなど）やルイス塩基（例えば、トリフェニルホスフィンなどの3価リン化合物）などが挙げられる。また重合禁止剤としては、ハイドロキノンやフェノチアジンなどが挙げられる。

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【化3】



(ただし、nは0以上の整数を示す)

【0009】本発明のエポキシアクリレート樹脂は、感光性材料として、単独ではもちろん他のベース樹脂や反応性希釈剤と組み合わせて使用することにより、各種コーティング剤（特に耐熱性を必要とする用途など）、UV塗料、あるいはレジスト用途など幅広い用途に使用することができる。

【0010】

【実施例】本発明を以下の実施例により説明するが、本発明はこれらに限定されるものではない。尚、実施例中、部と表示してあるのは、全て重量部を示す。

【0011】実施例 1

前記式(2)のエポキシ樹脂(エポキシ当量177g/eqiv.) 354部とアクリル酸140部を60℃で溶解させた後、触媒(塩化トリエチルベンジル) 3.4部と重合禁止剤(ハイドロキノン) 0.1部を添加し、90～95℃に加熱して14時間撹拌した。反応中、酸価とエポキシ当量を測定し、酸価2.0mg KOH/g、エポキシ当量15000g/eqiv. になるようアクリル酸または式(2)のエポキシ樹脂を添加し、酸価2.0mg KOH/g以下、エポキシ当量15000g/eqiv. 以上になるまで加熱撹拌を続けた。測定値が目標に達した後、60℃まで冷却し、目的物を得た(収率98%)。この目的物は、60℃で粘稠な液体であった。

【0012】比較例 1

実施例1のエポキシ樹脂354部の代わりにビスフェノールAジグリシジルエーテル(エポキシ当量180g/eqiv.) 360部を用い、同様に合成して、目的物を得た(収率97%)。この目的物は、60℃で粘稠な液体であった。

【0013】比較例 2

実施例1のエポキシ樹脂354部の代わりにナフタレンジグリシジルエーテル(252g/eqiv.) 500部を用い、同様に合成して、目的物を得た(収率97

%)。この目的物は80℃で粘稠な液体であった。

【0014】実施例1、比較例1、2で得られた樹脂について下記特性の比較評価を行なった。

感光性の評価

各試料100部にDarocure 1173(チバガイギー製、1-フェニル-2-ヒドロキシ-2-メチルプロパン-1-オン) 3部を混練し、スピンコーターにより膜厚50μmのフィルムを作成した。これを、高圧水銀灯を用いて300～600mj/cm²の光量で露光した。UV硬化したものは○、硬化しないものは×として表示した。

【0015】尚、比較のために用いた樹脂のうち、比較例2(ナフタレン骨格含有エポキシアクリレート)は、単独でのUV硬化が困難であったため、以下の物性の比較評価は、全て次の方法で作成した試料を用いた。即ち、実施例1、比較例1、2で得た各試料100部にバブチルZ(日本油脂(株)製、1-ブチルパーオキシベンゾエート) 2部を混練し、スピンコーターにより膜厚50μmのフィルムを作成し、これを窒素気流下、150℃・2時間で硬化した。

【0016】耐熱性(TG(℃) 10%減量温度)の評価

各試料の硬化膜の熱重量分析(TG)を行ない、10%減量温度を求めた。

【0017】耐熱性(ガラス転移点(℃) 1Hz)の評価

各試料の硬化膜の動的粘弾性測定を行ない、これにより、ガラス転移点を求めた。また、各試料の弾性率の温度依存性チャートを図1～図3に示した。

【0018】吸水率(%)の評価

各試料の硬化膜を、恒温恒湿槽中、85℃・85%で24時間放置し、前後の重量変化から吸水率を測定した。

【0019】以上の物性の評価結果を表1に示す。

【表1】

表 1

	実施例1 式 (1) の化合物	比較例1 ビスフェノールAジグリシ ルエーテルアクリレート	比較例2 ナフタレンジグリシル エーテルアクリレート
感光性	○	○	×
TG (℃) (10% 減量温度)	380	370	370
ガラス転移点 (℃) 1Hz	171	159	138
吸水率 (%)	0.8	1.6	1.4

【0020】表1の結果から本発明による式(1)の化合物は、汎用エポキシアクリレート(ビスフェノールA型)や配向性の高いエポキシアクリレート(ナフタレン骨格含有型)と比較して、高いガラス転移温度と低い吸水率を有することがわかる。また図1～図3の弾性率の温度依存性チャートから、本発明による式(1)の化合物は、ガラス転移点前後の弾性率の変化が小さく、従って内部応力の発生の低い硬化物であることがわかる。

【0021】

【発明の効果】以上詳述したように、本発明の新規なエポキシ(メタ)アクリレート樹脂は、近年の要求特性の高度化に充分に対応できる高機能性高分子材料として極めて有用であり、熱的、化学的に安定な優れた高分子材

*料として、各種コーティング剤(特に耐熱用途)、UV塗料、レジスト用途などの幅広い用途に使用することができる。

【図面の簡単な説明】

【図1】式(1)の化合物を使用した硬化物の弾性率の温度依存性チャートを示したものである。

【図2】ビスフェノールAジグリシルエーテルアクリレートを使用した硬化物の弾性率の温度依存性チャートを示したものである。

【図3】ナフタレンジグリシルエーテルアクリレートを使用した硬化物の弾性率の温度依存性チャートを示したものである。

【図1】

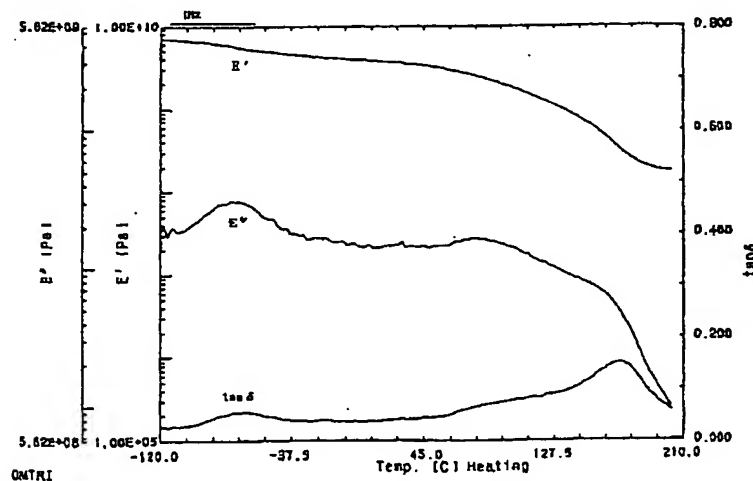


図1 式(1)の化合物

【図2】

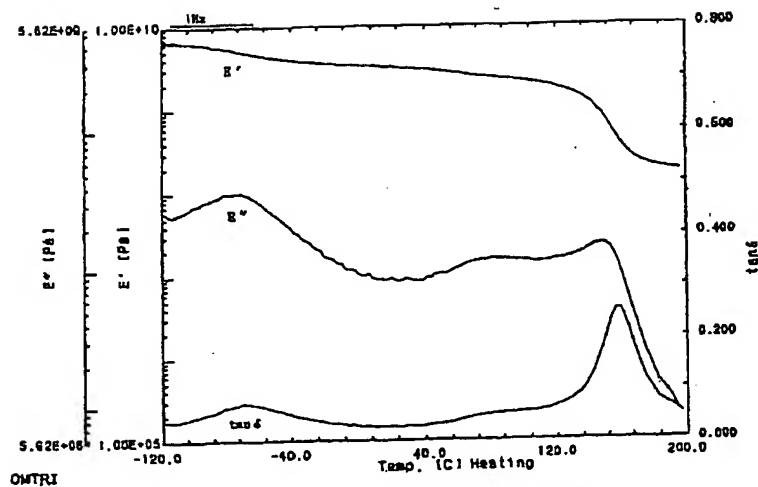


図2 ビスフェノールAジグリシジルエーテルアクリレート

【図3】

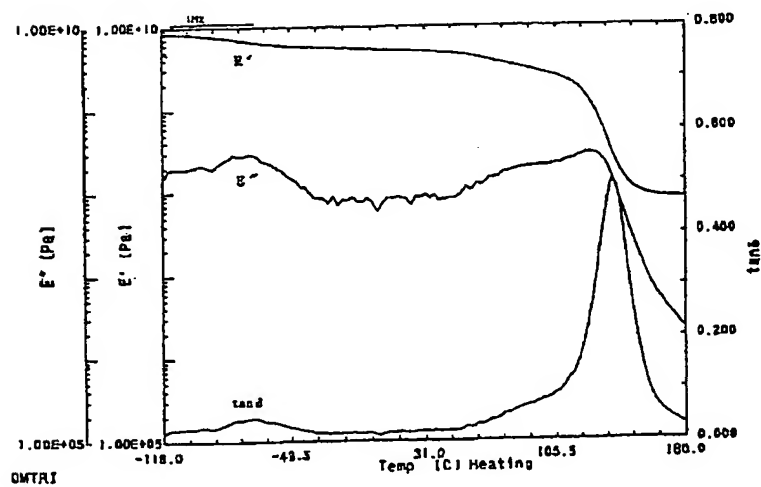


図3 ナフタレンジグリシジルエーテルアクリレート

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-157340

(43)Date of publication of application : 17.06.1997

(51)Int.Cl.

C08F299/00

C09D 4/02

(21)Application number : 07-345682

(71)Applicant : KYOEISHA CHEM CO LTD

(22)Date of filing : 08.12.1995

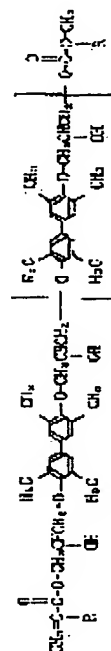
(72)Inventor : KUKI YUKIHIRO
TAKENAKA NAOI

(54) NOVEL HEAT-RESISTANT RESIN

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a novel heat-resistant resin which has a high glass transition temp. and low moisture absorption and is less likely to create stress and tough by using an epoxy (meth)acrylate resin (BE) having a biphenyl skeleton as a functional polymer material.

SOLUTION: The BE resin is represented by formula I (wherein R represents H or methyl; and (n) is an integer of not less than 0) and prepd. by a general epoxy synthetic process. Specifically, it is easily prepd., in the absence of a solvent, by reacting an epoxy resin, with both terminals being glycidyl etherified, with (meth) acrylic acid in the presence of a catalyst (e.g. a quaternary salt or a Lewis base with specific examples thereof including triethylbenzyl chloride) and a polymn. inhibitor (e.g. hydroquinone) at 90 to 100° C for 12 to 15hr. The BE resin may be used, as a photosensitive material, either alone or in combination with other base resin(s) and a reactive diluent for applications such as various coating agents (particularly applications where heat resistance is required), UV coating materials, or resists.



LEGAL STATUS

[Date of request for examination] 05.10.1998

[Date of sending the examiner's decision of rejection] 20.02.2001

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's
decision of rejection]

[Date of extinction of right]

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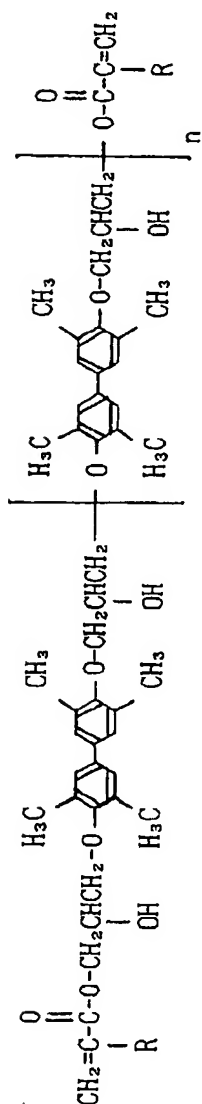
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 CLAIMS

[Claim(s)]

[Claim 1] Epoxy (meta) acrylate resin which has the biphenyl frame expressed with the following type (1): [Formula 1]

(1)



(However, R in a formula expresses H or CH₂, and n shows zero or more integers.)

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention is especially useful about new epoxy (meta) acrylate resin excellent in thermal resistance, photosensitivity, and low hygroscopicity as the base resin for a solder resist or plating resists, or various resin for coatings. Furthermore, it can use for various applications as stable outstanding polymeric materials thermally and chemically by using it, blending with the compound which the epoxy (meta) acrylate resin of this invention accepts not only in these, and independent accepts the need from the first, and has various photosensitivity.

[0002]

[Description of the Prior Art] Conventionally, epoxy acrylate resin has been used in broad fields, such as a cross linking agent of the charge of sensitization material as various coating materials, the charge of optical material, dental materials, and a macromolecule. However, the physical properties searched for as a functional polymer material are becoming still severer with the advancement of the demand characteristics in these applicable fields in recent years. As these physical properties, although thermal resistance, weatherability, low hygroscopicity, a high refractive index, high fracture toughness, etc. are called for, these demand physical properties have not necessarily been satisfied the place which is the former.

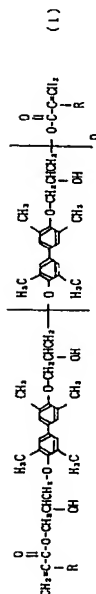
[0003] A bisphenol type thing is mentioned as epoxy (meta) acrylate used the general-purpose in the field of the charge of sensitization material used now. About a low-molecular-weight type (for example, bisphenol A diglycidyl ether diacrylate), although this bisphenol type of epoxy acrylate has a comparatively high glass transition temperature, the top where water absorption is high has the fault of being easy to generate internal stress. Although what is necessary is to enlarge molecular weight generally and just to lower crosslinking density, in order to suppress water absorption and to control generating of stress, since glass transition temperature falls conversely, there is a limitation also in the approach of starting. Moreover, the present condition is that there are merits and demerits about physical properties about the epoxy acrylate of other types as well as the above. Although various epoxy resin raw materials which improve thermal resistance and water absorption are being developed in view of this present condition recently, it will not have resulted, by the time all can satisfy all the reduction of thermal resistance, water absorption, and stress.

[0004]

[Problem(s) to be Solved by the Invention] It is originated in order to cancel the trouble of this conventional technique, and the place made into the purpose has this invention in offering the new epoxy (meta) acrylate resin which can be satisfied with a Prior art of all the high demand characteristics that were not able to be attained. Although high thermal stability is mentioned as said demand characteristics and a high glass transition temperature is needed in the first place for that purpose, about this, it can attain by raising the crosslinking density of resin. Moreover, if glass transition temperature is made high, the stability in an elevated temperature will be maintained, but in this case, if an evaporation component mixes in the interior of resin, it will become the cause of a crack. Since an evaporation component is especially included automatically if the water absorption of resin is high, the inclination becomes strong. Therefore,

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(However, R in a formula expresses H or CH₂, and n shows zero or more integers.)

[0008]

[Embodiment of the Invention] The epoxy (meta) acrylate resin of this invention can obtain easily under a non-solvent the epoxy resin, acrylic acid, or methacrylic acid which is shown for example, by the following formula (2), which is obtained by the general epoxy resin synthesis method and with which both ends were glycidyl-ether-ized under existence of a catalyst and polymerization inhibitor by making it react at 90-100 degrees C for 12 to 15 hours. As a catalyst, the 4th class salts (for example, tetra-alkylammonium halide, dihalo triphenyl phosphorane, etc.), a Lewis base (for example, trivalent phosphorus compounds, such as triphenyl phosphine), etc. are mentioned. Moreover, hydroquinone, phenothiazin, etc. are mentioned as polymerization inhibitor.

[Formula 3]

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unless very severe humidity management is carried out, it becomes impossible to use hygroscopic high resin, and it becomes indispensable [low hygroscopicity] to thermal stability. Furthermore, since it becomes weak so that the internal stress by thermal expansion is large, reduction of stress is also an important element.

[0005] As mentioned above, in order to raise glass transition temperature, generally the crosslinking density of resin will be raised, but if crosslinking density is raised, the inclination for water absorption to rise will be accepted, as the technique of raising glass transition temperature, while controlling water absorption — — — introducing the frame which has a high substituent (for example, polycyclic type ring) and a high stacking tendency is known. However, since it is easy to generate stress, such resin is hard and weak. Therefore, it has a high glass transition temperature, and it is low hygroscopicity and development of the tough highly efficient nature epoxy acrylate resin which stress moreover cannot generate easily is demanded.

[0006]

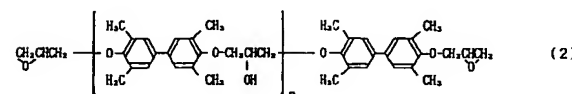
[Means for Solving the Problem] The epoxy (meta) acrylate resin shown in the following formula

(1) has a high glass transition temperature, and carried out the knowledge of being low hygroscopicity and being tough highly efficient nature epoxy acrylate resin which stress moreover cannot generate easily, and this invention persons resulted in completion of this invention, as a result of inquiring wholeheartedly, in order to attain the above-mentioned purpose.

[0007] namely, : whose this invention is epoxy (meta) acrylate resin which has the biphenyl frame expressed with the following type (1) — [Formula 2]

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(However, n shows zero or more integers)

[0009] As a photosensitive ingredient, if the epoxy acrylate resin of this invention is independent, it can be used for broad applications, such as various coating agents (application which needs especially thermal resistance), UV coating, or a resist application, by using it combining other natural base resin or a reactant diluent.

[0010]

[Example] Although the following examples explain this invention, this invention is not limited to these. In addition, all having displayed it as the section show the weight section among an example.

[0011] Example After dissolving the epoxy resin (weight-per-epoxy-equivalent 177g/eqv.) 354 section of the 1 aforementioned type (2), and the acrylic acid 140 section at 60 degrees C, the catalyst (chlorination triethyl benzyl) 3.4 section and the polymerization inhibitor (hydroquinone) 0.1 section were added, and it heated at 90-95 degrees C, and agitated for 14 hours. The acid number and weight per epoxy equivalent were measured during the reaction, the acrylic acid or the epoxy resin of a formula (2) was added so that it might become acid-number 2.0 mgKOH/g and weight-per-epoxy-equivalent 15000 g/eqv., and heating churning was continued until it became more than 2.0 or less mgKOH/g of acid numbers, and weight-per-epoxy-equivalent 15000 g/eqv. After measured value reached the target, it cooled to 60 degrees C and the specified substance was obtained (98% of yield). This specified substance was a viscous liquid at 60 degrees C.

[0012] Example of a comparison The bisphenol A diglycidyl ether (weight-per-epoxy-equivalent 180g/eqv.) 380 section was used instead of the epoxy resin 354 section of one example 1, it compounded similarly, and the specified substance was obtained (97% of yield). This specified substance was a viscous liquid at 60 degrees C.

[0013] Example of a comparison The naphthalene diglycidyl ether (252g/eqv.) 500 section was used instead of the epoxy resin 354 section of two examples 1, it compounded similarly, and the specified substance was obtained (97% of yield). This specified substance was a viscous liquid at 80 degrees C.

[0014] Comparative evaluation of the following property was performed about the resin obtained in an example 1 and the examples 1 and 2 of a comparison.

It is Darocure to evaluation each sample 100 photosensitive section. The 1173(Ciba-Geigy make, 1-phenyl-2-hydroxy-isobutane-1-ON) 3 section was kneaded, and the film of 50 micrometers of thickness was created by the spin coater. A high pressure mercury vapor lamp is used for this, and it is 300 - 800 mJ/cm². It exposed with the quantity of light. What carried out UV hardening displayed as x O and the thing which is not hardened.

[0015] In addition, since UV hardening independently was difficult for the example 2 (naphthalene frame content epoxy acrylate) of a comparison among the resin used for the comparison, all of the comparative evaluation of the following physical properties used the sample created by the following approach. Namely, the par butyl Z(Nippon Oil & Fats Co., Ltd. make, 1-butyl-peroxy benzoate)2 section was kneaded in each sample 100 section obtained in an example 1 and the examples 1 and 2 of a comparison, the film of 50 micrometers of thickness was created by the spin coater, and this was hardened under the nitrogen air current in 150 degree C and 2 hours.

[0016] Thermogravimetric analysis (TG) of the hardening film of evaluation each heat-resistant (TG(degree-C) 10% loss-in-quantity temperature) sample was performed, and loss-in-quantity temperature was searched for 10%.

[0017] Dynamic viscoelasticity measurement of the hardening film of evaluation each heat-

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resistant (glass transition point (degree C) 1Hz) sample was performed, and this asked for the glass transition point. Moreover, the temperature dependence chart of the modulus of elasticity of each sample was shown in drawing 1 - drawing 3.

[0016] the hardening film of evaluation each sample of water absorption (%) — constant temperature — it was left at 85 degrees C and 85% among the constant humidity chamber for 24 hours, and water absorption was measured from a weight change [before and after].

[0018] The evaluation result of the above physical properties is shown in Table 1.

[Table 1]

表 1

	実施例1 式 (1)の化合物	比較例1 CJ-J-4LS799 5LS-79779-t	比較例2 779-79795LS 2-7979-t
感光性	○	○	×
TG (°C) (10% 減重温度)	380	370	370
T _g 転移点 (°C) 1Hz	171	159	138
吸水率 (%)	0.8	1.6	1.4

[0020] The result of Table 1 shows that the compound of the formula (1) by this invention has a high glass transition temperature and low water absorption as compared with general-purpose epoxy acrylate (bisphenol A mold) or the high epoxy acrylate (naphthalene frame content mold) of a stacking tendency. Moreover, the temperature dependence chart of the modulus of elasticity of drawing 1 - drawing 3 shows that the compound of the formula (1) by this invention is a low hardened material of generating of internal stress small [change of the modulus of elasticity before and behind a glass transition point] therefore.

[0021]

[Effect of the Invention] As explained in full detail above, the new epoxy (meta) acrylate resin of this invention is very useful as highly efficient nature polymeric materials which can fully respond to the advancement of demand characteristics in recent years, and can be used for an application with broad various coating agents (especially heat-resistant application), UV coating, resist application, etc. as stable outstanding polymeric materials thermally and chemically.

[Translation done.]

* NOTICES *

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- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.*** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The temperature dependence chart of the modulus of elasticity of the hardened material which used the compound of a formula (1) is shown.

[Drawing 2] The temperature dependence chart of the modulus of elasticity of the hardened material which used bisphenol A JIKURISHIJIRU ether acrylate is shown.

[Drawing 3] The temperature dependence chart of the modulus of elasticity of the hardened material which used naphthalene diglycidyl ether acrylate is shown.

[Translation done.]